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**Assessment of Some Advanced Protective Schemes,
Including Chromate and Non-Chromate Conversion
Coatings for Mg Alloy ZE41A-T5 Using
Electrochemical Impedance Spectroscopy**

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ABSTRACT

A study has been conducted to evaluate the suitability of a non-chromate conversion coating (Tagnite 8200) for replacing the currently used chromate conversion coating (Dow 17) on Mg alloy ZE41A-T5. This replacement must be accomplished without compromising the corrosion resistance and protection efficacy of other advanced coating schemes. Electrochemical Impedance Spectroscopy (EIS) and salt spray tests have been employed to compare the corrosion behavior in chloride containing solutions of Mg alloy ZE41A-T5 which has been coated with various combinations of a conversion coating (Tagnite 8200 or Dow 17), primer (Sermetel 1083 or epoxy) and topcoat (Sermetel 1089, polyurethane, or epoxy electrodeposited E-coat). Results indicate that there are several coating schemes which perform better than the currently used system and that the EIS and the salt spray tests show a direct correlation.

INTRODUCTION

Magnesium alloys are known for their low specific weight and excellent mechanical properties, thus they are considered to be one of the best structural materials for aircraft applications. However, U.S. Army experience with magnesium helicopter components has shown that significant corrosion problems exist which require increased maintenance, impacting both cost and readiness. During the Vietnam era magnesium alloys were used widely to reduce weight and increase performance. But in a recent modernization program, a number of magnesium parts were replaced with aluminum alloys

to reduce corrosion problems, resulting in a concomitant weight penalty. It is clear that improved protective schemes are needed to provide corrosion resistant magnesium components before its inherent weight advantage can be fully utilized.

The current practice for protecting magnesium employs an anodizing chromate conversion pretreatment, an epoxy primer and a polyurethane topcoat. Previously, we described¹ the beneficial results achieved by interposing a baked epoxy resin sealer between the conversion coating and primer application. In a recent paper², we reported some of our results on the corrosion resistance of magnesium alloys ZE41A-T5 and WE43A-T6 which were subjected to various coating schemes. These results were compared to the corrosion resistance of the current standard coating used at Sikorsky. In this paper, we present additional results obtained from ZE41A-T5 alloy panels with similar protective schemes, but replacing the Dow 17 surface pretreatment (with chromates) with Tagnite 8200 (non-chromate). Since the Tagnite is chromate free, it is considered to be more environmentally acceptable. However, the protection efficacy of various coating schemes in combination with the Tagnite pretreatment must be investigated prior to its qualification as a replacement for Dow 17.

MATERIALS

Magnesium alloy ZE41A-T5 (UNSM16410) was selected because it is being used in newer aircraft. Table 1 contains the mechanical properties and the nominal composition in wt% of elements. Each coating has been given a letter designation which is described in Table 2. Table 3 lists each of the evaluated protective schemes and thicknesses.

The Sermetel 1083 primer (SP) is a high temperature polymer coating formulated with metallic pigmentation and corrosion inhibitors. This primer is applicable over conversion (anodized or immersion) pretreated surfaces such

Table 1
Mechanical Properties of ZE41A with Nominal Compositions in Wt% Solute

Alloy	Mechanical Properties						Composition				
	UTS	1% YS	2% YS	Modulus	%RA	%EL	Zn	RE	Zr	Y	Mg
ZE41A T-5(Ksi)	31.40	20.80	22.30	6520	4.30	4.80	4.3	1.5	0.73	-	93.47
ZE41A T-5(MPa)	216.50	143.42	153.76	44960							

Table 2
Letter Designations for Protective Schemes

Coating Designation	Pretreatment	Primer	Topcoat	Notes
dSP	Dow 17	Sermetel 1083		Sermetel Primer
dSPT	Dow 17	Sermetel 1083	Sermetel 1089	Sermetel Primer + Topcoat
dSk	Dow 17	MIL-P-85582	MIL-C-46168	Sikorsky (Standard)
tSP	Tagnite 8200	Sermetel 1083		Sermetel Primer
tSPT	Tagnite 8200	Sermetel 1083	Sermetel 1089	Sermetel Primer + Topcoat
tSPn	Tagnite 8200	Sermetel 1321		Sermetel Non-Cr Primer
tE	Tagnite	Epoxy E-Coat		E-Coat

Table 3
Protective Schemes Evaluated by US Army Research Laboratory

Substrate	Anodic Pre-Treatment (Thickness)		Coating System (Thickness)
	Dow 17	Tagnite	
ZE41A-T5			
✓	✓	✓	SP- (1mil, 2mil)
✓	✓	✓	SPT- (1mil, 2mil)
✓	✓		SP- (1mil) +Sk
✓	✓		SP- (2mil) +Sk
✓	✓		Sk
✓	✓		SPn (1 mil)
✓		✓ (0.35mil)	
✓		✓ (0.80mil)	
✓		✓ (0.90mil)	(phosphate sealed)
✓		✓ (1.0mil)	(phosphate sealed) +E
✓		✓ (0.30mil)	E- (0.80mil)
✓		✓	SPn (1 mil)

as Dow 17 (Dow Chemical Co. anodized, MIL-M-45202B, type II, class D, $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ sodium dichromate) and Tagnite 8200 (non-chromate, Technology Application Group, Inc.). This primer is also compatible with a variety of topcoats, including Sermetel 1089 (T) a high temperature polymer and the currently used system by Sikorsky (Sk) which incorporates both an epoxy VOC compliant primer (MIL-P-85582) and a polyurethane topcoat (MIL-C-46168, chemical agent resistant topcoat, CARC). Tagnite 8200 is a two step (non-chromate anodizing and immersion) surface pretreatment. It contains silicon oxide and can be topcoated with an epoxy sealer/primer/topcoat combination such as SP, SPT, or electrodeposited epoxy E-coat (E).

EXPERIMENTAL

The testing program included both exposure to salt spray (ASTM

B117) and immersion in 0.5N NaCl solution, open to the air, for electrochemical impedance spectroscopy (EIS) measurements. EIS has been used extensively for assessing coating protection efficacy at the Army Research Laboratory (ARL) ¹⁻³. In salt spray testing, the specimens were visually examined periodically for corrosion. Testing was terminated if a panel was found to have corroded through its coating thickness. In most cases, only one panel for each test with each coating scheme was tested due to a limited number of coated panels available.

The cell used for EIS testing has been described elsewhere¹. EIS tests were performed with a PAR 378 system consisting of a model 5208 two-phase lock-in analyzer, a model 273 potentiostat/galvanostat, and a IBM PC XT computer. Periodic measurements were taken for the samples exposed to 0.5N NaCl solution at the corrosion potential (stabilized within 1 hour) over the frequency range of 100 KHz-0.005 Hz for a period of up to 100 days at room temperature. In the initial two or three weeks of immersion, EIS measurements were conducted more frequently since large variations were expected to occur; at longer times readings were taken less frequently since the impedance had stabilized. The single sine technique with an input sinusoidal potential of 5mV was applied in the frequency range 100KHz -5 Hz. At the lower frequency of 10-0.005 Hz, the multi-sine technique was employed with an input sinusoidal voltage of 10 mV. The dimensions of the magnesium alloy test panels were 6"x4"x1/4" (15.2cm x 10.2cm x 0.64cm). They were coated by various vendors on both front and back surfaces as described in Table 2. EIS tests were conducted only on the coated machined surface of the panels. EIS tests were performed in duplicate with results expressed as an average of the two panels or two different areas on the same panel due to panel availability. Before testing, the coated surfaces were cleaned with compressed filtered air and only areas without visually detectable defects were selected for exposure to chloride solutions.

The collected data was plotted and evaluated in the Bode as well as the Nyquist formats. It was found that the Bode formats were easier to analyze, and thus more useful in terms of determining coating efficacy. The Bode formats display the magnitude ($\log|Z|$) and phase angle (Θ) of the impedance as a function of applied frequency ($\log f$). The total impedance of the specimen, defined as the $\log|Z|$ value extrapolated to 1 mHz in the Bode magnitude plot, was also plotted as a function of exposure time for comparison of the efficacy of each coating system.

RESULTS AND DISCUSSION

Salt Spray

Table 4 contains ZE41A-T5 salt spray data for the various protective schemes. These schemes are comprised of either the Dow 17 (d) or the Tagnite (t) pretreatment, then coated with either the primer only (SP), primer and topcoat (SPT), or with the Sikorsky (Sk) system. Included also is a Tagnite/sealer/electrodeposited epoxy E coat system(tE) and a proprietary non-Cr primer, Sermetel 1321 (tSPn).

Table 4
Salt Fog Performance Summary of Coated ZE41A-T5
Dow 17

Hours	Coating dSPT (1 mil)	Coating dSP (1 mil) + Coating Sk	Coating dSPT (2 mil)	Coating dSP (2 mil) + Coating Sk
% Area Failed				
6	0	0	0	0
24	0	0	0	0
5424	10	50	0	0
6024	Failed	Failed	0	0
6696			0	0
14976			0	0
↓			↓	↓
19440			0	0

Tagnite 8200

Hours	Coating tSPT (1 mil)	Coating tSP (1 mil)	Coating tSPT (2 mil)	Coating tSP (2 mil)	Coating tSPn (1 mil) + 1321
% Area Failed					
6	0	0	0	0	0
720	0	0	0	0	0
1200	0	4	0	0	0
1632	0	7	0	0	0
1944	0	7	0	0	0
2532	0	10	0	0	0
3384	0	10	0	0	0
3624	0	12	0	0	0
4224	0	15	0	0	0
4368	0	18	0	0	0
4560	0	20	0	0	0
4872	0	22	0	0	0
4968	0	25 - Failed	0	0	0
5376	0		0	0	0
5664	0		0	0	0
6168	0		0	0	0
6480	5		0	0	0
6840	10 - Failed		5	7	0

Note: Failures of ZE41A panels were defined by non-edge nucleating pits which corroded through the entire thickness of the test panel.

All panels pretreated with Dow 17 and coated with various schemes

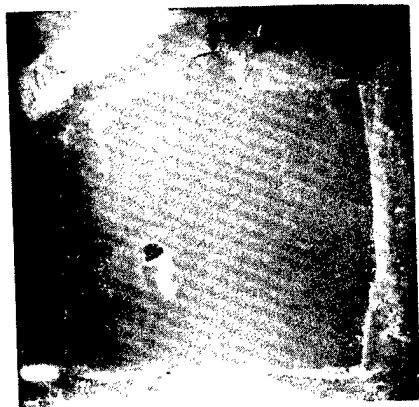
completed at least 5000 hours of salt spray exposure without failure. However, for coatings with only 1 mil (.0254mm) of either the primer (dSP) or the primer/topcoat combination (dSPT), corrosion occurred through the thickness shortly thereafter at 6024 hours. When the total thickness of these coatings was increased to 2 mils (.0508 mm), they performed significantly better. No failure occurred up to 15,000 hours of exposure, yielding a 3X improvement.

For those panels pretreated with Tagnite and coated with various coating schemes, failure was defined as corrosion of a panel through its entire thickness at a non-edge nucleation site. Edge related corrosion attacks were ignored since they resulted from improper sealing along the edges. The panel coated with 1 mil total of tSP was first to fail at 4968 hours followed by the panel coated with 1 mil total of tSPT at 6840 hours. The panels coated with 2 mil total coatings contained some small corroded pits or blisters, but no failures occurred at 6840 hours. These results indicate that the corrosion resistance of the coated panels are more affected by coating thickness regardless of the type of pretreatment employed. It is also shown that the corrosion resistance of the coated samples appear to be comparable if the coating schemes and their total thickness are the same. The same may be said based on their EIS results as presented later. Additional ARL salt spray data for the Tagnite pretreated samples is unavailable due to the limited number of panels provided. However, Sikorsky⁴ did conduct 96 hours of salt spray testing (ASTM B117) on magnesium alloy panels pretreated with Dow 17, HAE (non-Cr type), or Tagnite only. Their results showed that Tagnite provided greater protection with only minor corrosion occurring as compared to the extensive corrosion observed on the other samples.

It was noted that the panel coated with a 1 mil non-Cr primer, Sermetel 1321 (tSPn), showed no corrosion occurring at 6840 hours. This non-Cr primer appears to be a potential candidate for the replacement of the Cr containing Sermetel 1083 (SP). Additional results will be reported at a later date since the testing is still in progress.

Figure 1(a-c) shows the front and back of tested panels coated with 1 mil dSPT, 1 mil tSPT, and 2 mils tSPT respectively. In the 1 mil specimens, the non-edge nucleated corrosion has proceeded through the thickness of the substrate after having been subjected to at least 5,000 hours of salt spray testing. The corroded area appearing on the front surface is small but spreads quickly, exhibiting much broader corrosion on the rear surface. The non-edge nucleated corrosion has not occurred on the panel coated with 2 mil tSPT.

as shown in Figure 1c.



(a)

Front



Back

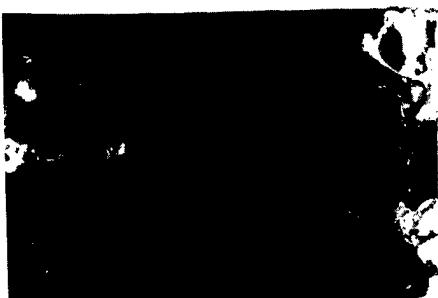


(b)

Front



Back



(c)

Front



Back

Figure 1. Front and Back of Failed Salt Spray Tested Specimens; a) 1 mil dSPT Failed at 5,000 hours, b) 1 mil tSPT Failed at 6840 hours, c) 2 mil tSPT Unfailed at 6840 hours,

Electrochemical Impedance Spectroscopy

The EIS data obtained from each test was plotted in both Bode and Nyquist formats. Certain parameters were examined to ascertain whether they might be used to characterize protection efficacy of the coatings. Representative plots are shown in Figure 2. Impedance values at low frequency ($|Z|$ @ 1 mHz) can distinguish between good and poor coatings⁵. For example⁶, for aluminum alloys, any coating with total impedance values $<10^5 \Omega \cdot \text{cm}^2$ indicates repainting is necessary; values between 10^6 and $10^8 \Omega \cdot \text{cm}^2$ indicate reexamination is required in 6 months to a year; when values $>10^9 \Omega \cdot \text{cm}^2$ are measured, the coating is functioning properly. Murray and Hack⁷ accumulated EIS data over a frequency range of 5×10^{-3} to 10^5 Hz for relatively thick MIL-P-24441 epoxy coated steel specimens which were exposed to artificial ocean water for up to 3 years. They found that the most useful parameter is Z_{max} , defined as the maximum impedance at low frequency. Samples with Z_{max} values greater than $10^9 \Omega \cdot \text{cm}^2$ were characterized as superior coatings. (Note that a factor of 5.06 cm^2 should be multiplied to the impedance magnitude shown in the figures to obtain comparable $\Omega \cdot \text{cm}^2$ values.)

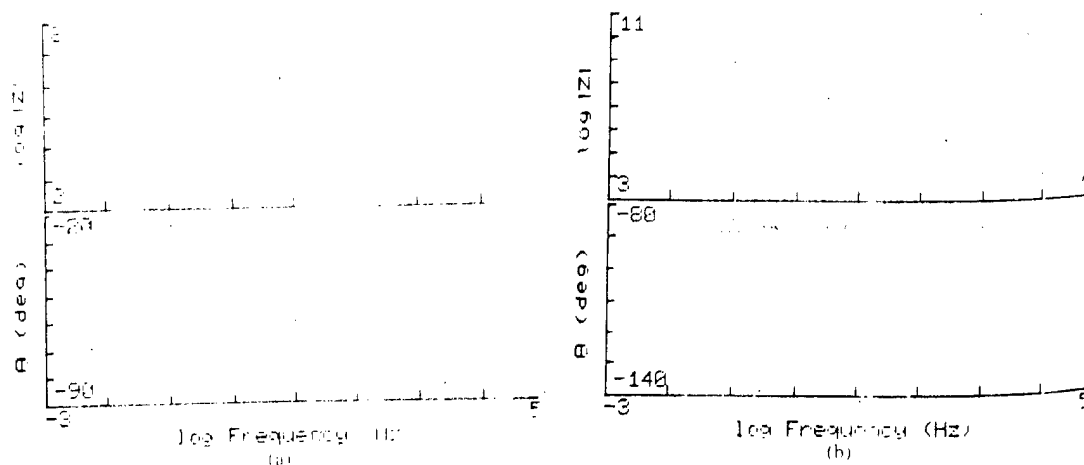


Figure 2. EIS Data Plotted in Bode Format a) 1 mil dSP , b) 1 mil tSP with 0.8 mil Coating E after 87 days of immersion in 0.5 N NaCl.

Figures 3 to 10 contain plots of impedance values extrapolated to 1 mHz as function of exposure time for the ZE41A-T5 coated samples described in Table 2.

Figure 3 compares the impedance of 1 mil thickness of coatings dSP, tSP, dSPT and tSPT. The beneficial effect of the Sermetel 1093 (T) barrier topcoat is shown on the Dow 17 (d) pretreated samples. The impedance of dSPT increased from $10^{9.5} \Omega$ to $> 10^{10} \Omega$ over the first 40 days. Although this value was higher than the impedance of the dSP at that time, the impedance of both samples was very much the same at the end of the test at 95 days, as seen in Figure 3(a). However, with the Tagnite (t) pretreated samples, Figure 3(b), the impedance data does not show the beneficial effect of the barrier topcoat. In the first 15 days, the impedance of the 1 mil tSP is higher than that of the 1 mil tSPT, $10^{9.5}$ vs. $10^{6.5} \Omega$. Afterwards, the impedance of both tSP and tSPT fluctuate between $10^{5.5}$ and $10^7 \Omega$ until the end of the test. Examination of samples after testing revealed that localized corrosion near the O-ring seal occurred in one of the 1 mil tSPT samples, resulting in lower impedance values, $\approx 10^5 \Omega$. The corrosion which occurred early in the test may have resulted from local O_2 content variation or pre-existing, macroscopic defects. Nevertheless, the application of a barrier topcoat layer does not appear to be beneficial at 1 mil total thickness since they both approach the same lower impedance values.

The comparison of the impedance of panels coated with 1 mil of Cr vs. non-Cr primer (Sermetel 1083 vs. Sermetel 1321) on Dow 17 (d) or Tagnite (t) is shown in Figure 4. It is clearly seen that the Tagnite/Sermetel 1321 shows a stable and higher impedance from the beginning to the end of the test period. This has been found to be in very good agreement with the results obtained from the salt spray test described previously. It also demonstrates that a good correlation exists between the EIS and salt spray test results qualitatively. Future tests have been planned to investigate further the compatibility of this non-Cr primer with other advanced coating schemes since it has exhibited excellent corrosion resistance in this initial study.

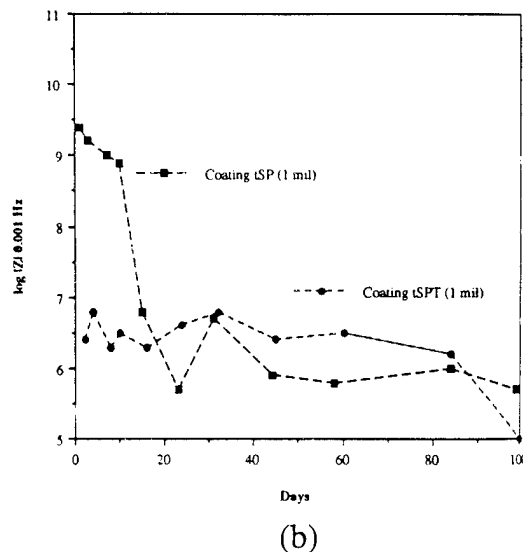
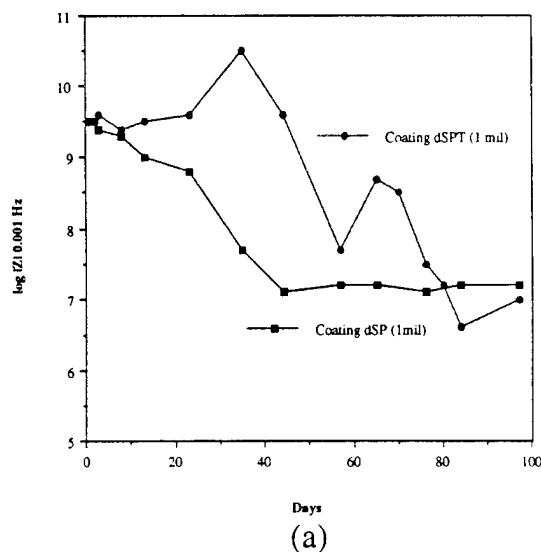


Figure 3. EIS data on 1 mil SP vs 1 mil SPT on ZE41A with
a) Dow 17 and b) Tagnite 8200

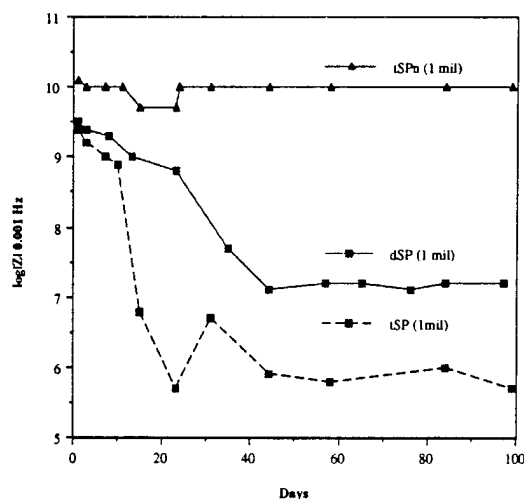


Figure 4. EIS data on 1 mil tSP, tSPn, and dSP.

Figure 5 shows the effect of coating thickness, 1 mil (25.4 mm) vs. 2 mil (50.8 mm) of SP on the performance of samples pretreated either by Dow 17(d) or by Tagnite (t). The impedances of the 2 mil (50.8mm) dSP or tSP coatings remained relatively constant, $>10^9 \Omega \cdot \text{cm}^2$, over a period up to 100 days, regardless of the type of pretreatment employed. This indicates that both are excellent coatings. The tSP coating also showed a similar magnitude

of impedance to the dSP coating over the test period. Generally, impedance is proportional to the thickness of the coating, since increased coating thickness restricts the corrosion inducing agents penetration to the substrate/coating interface by reducing the probability of a continuous defect pathway traversing through the thickness of the coating. Higher impedance values from EIS testing show the beneficial effect of increasing the thickness from 1

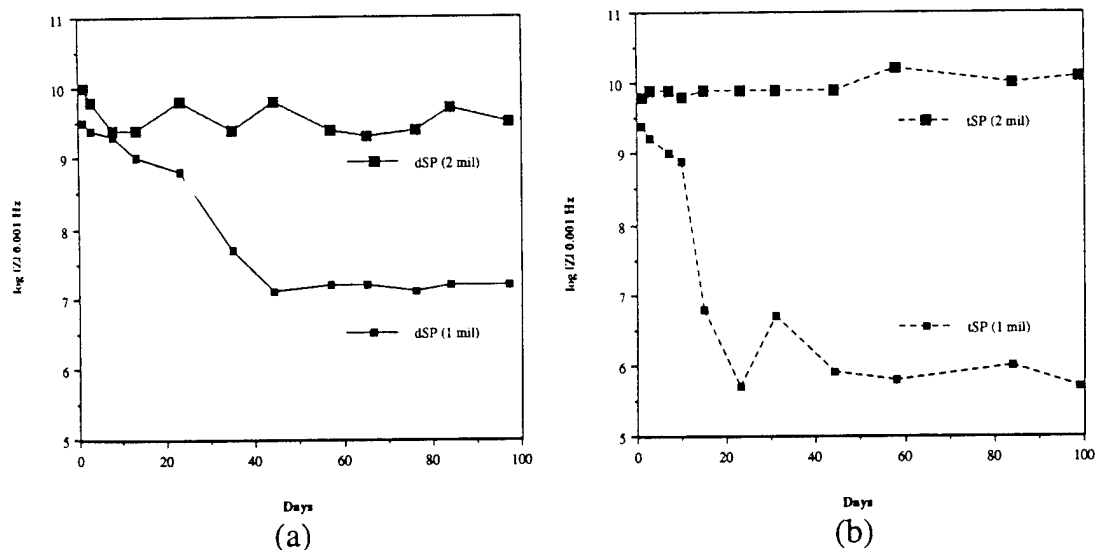


Figure 5. EIS data on 1 vs 2 mil SP on ZE41A with a) Dow 17 and b) Tagnite 8200.

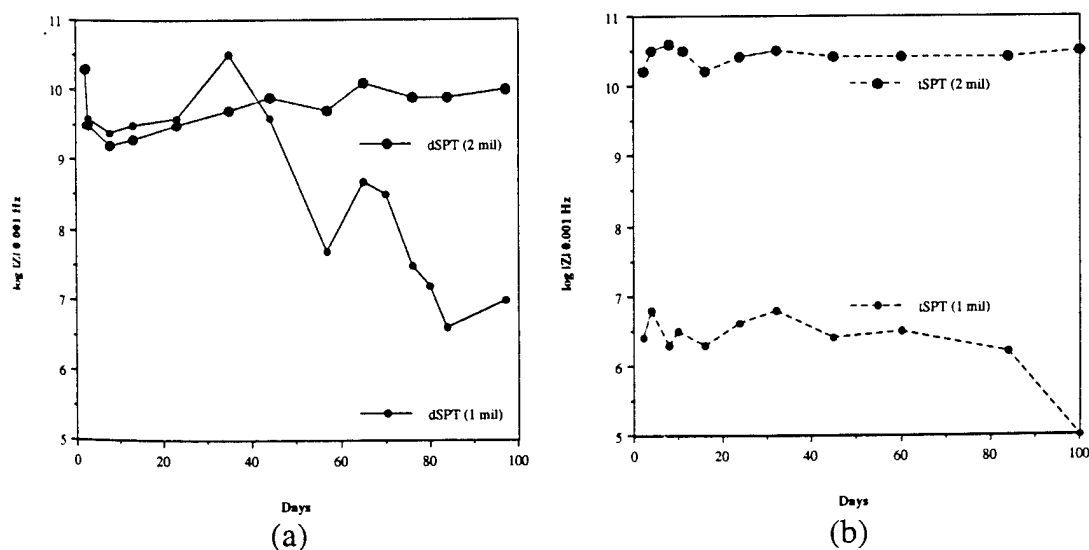


Figure 6. Effect of thickness, 1 vs 2 mil SPT on ZE41A with a) Dow 17 and b) Tagnite 8200.

to 2 mils of dSPT and tSPT. This is clearly shown in Figure 6, from 10^7 (dSPT) or from 10^5 (tSPT) to at least $10^{10} \Omega \cdot \text{cm}^2$ after 100 days of exposure to chloride solution.

When the total thickness is increased to 2 mil, the impedance of both coating systems, SP and SPT is comparable over the entire 100 day exposure period, with impedance values exceeding $10^9 \Omega \cdot \text{cm}^2$, as shown in Figure 7. The application of the barrier topcoat is beneficial to both the dSP and tSP coatings. For the dSP and dSPT coatings, Figure 7(a), impedance values begin to increase after 10 days following the initial drop. In the tSP and tSPT coatings, Figure 8(b), the impedance values show only slight temporal variation, with the 2 mil tSP having somewhat higher values than the tSP. It should be noted that these impedance values are of the same order of magnitude, indicating that the incremental effect of a barrier topcoat is much less significant than an increase in thickness.

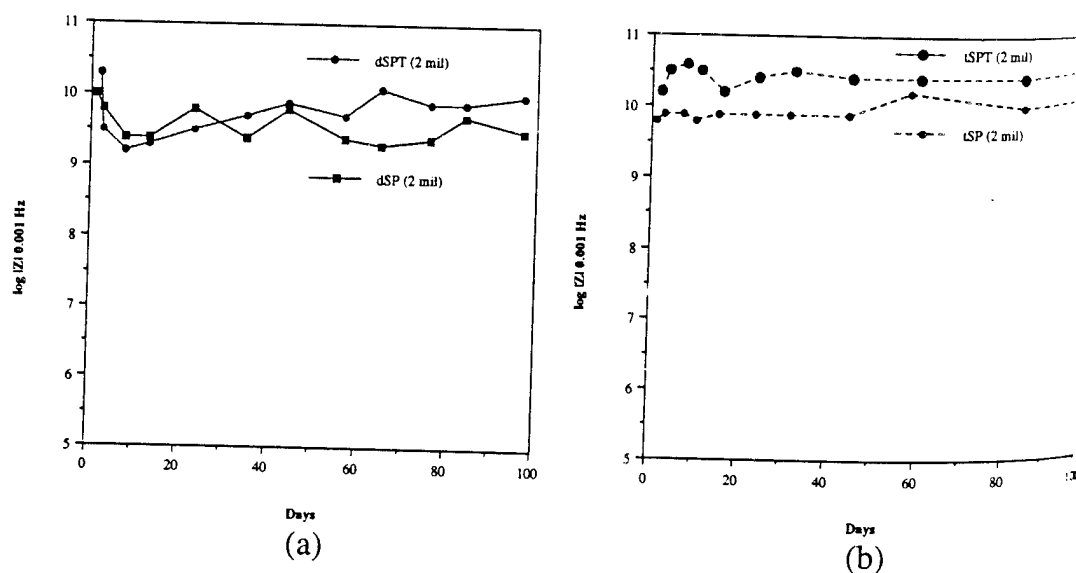


Figure 7. EIS data on 2 mil SP vs SPT on ZE41A with
a) Dow 17 and b) Tagnite 8200.

Figure 8 (a) compares the performance of coating dSP, at both 1 mil and 2 mils, with or without the currently used Sk coating system on top of it. The impedance of the currently used system, dSk (≈ 3 mil) is higher than that of the 1 mil dSP coating scheme, 10^8 vs. $10^7 \Omega \cdot \text{cm}^2$. It also remained relatively constant throughout the entire test while the latter scheme dropped dramatically over the first 41 days and remained constant thereafter. There is no further beneficial effect of applying the currently used Sk system on the top of the thicker 2 mil dSP coating system. In both cases impedance values were high and approximately the same, $10^{9.5} \Omega \cdot \text{cm}^2$. Figure 8(b) also

compares the impedance values of the currently used dSk coating with other schemes on top of Tagnite pretreated panels, including the 1 mil tSPn (non-Cr). It shows that the impedance of the 2 mil tSP and tSPT, as well as the 1 mil tSPn is consistently higher than the dSk over the test period of 100 days. However, similar to the data shown in Figure 8(a), the impedance of the 1 mil tSP and tSPT coatings are lower than that of the dSk system due to its thinness. For an impedance value of $10^9 \Omega \cdot \text{cm}^2$ over a 100 day testing period, a coating scheme minimum thickness of 2 mils is required to ensure its protection efficacy equal or greater than the currently used dSk scheme. There appears to be at least 6 coating systems; 2 mil dSP, 2 mil dSP/Sk, 2 mil dSPT, 2 mil tSP, 2 mil tSPT, 1 mil tSPn, which would meet this requirement, as shown in Figure 7 and 8.

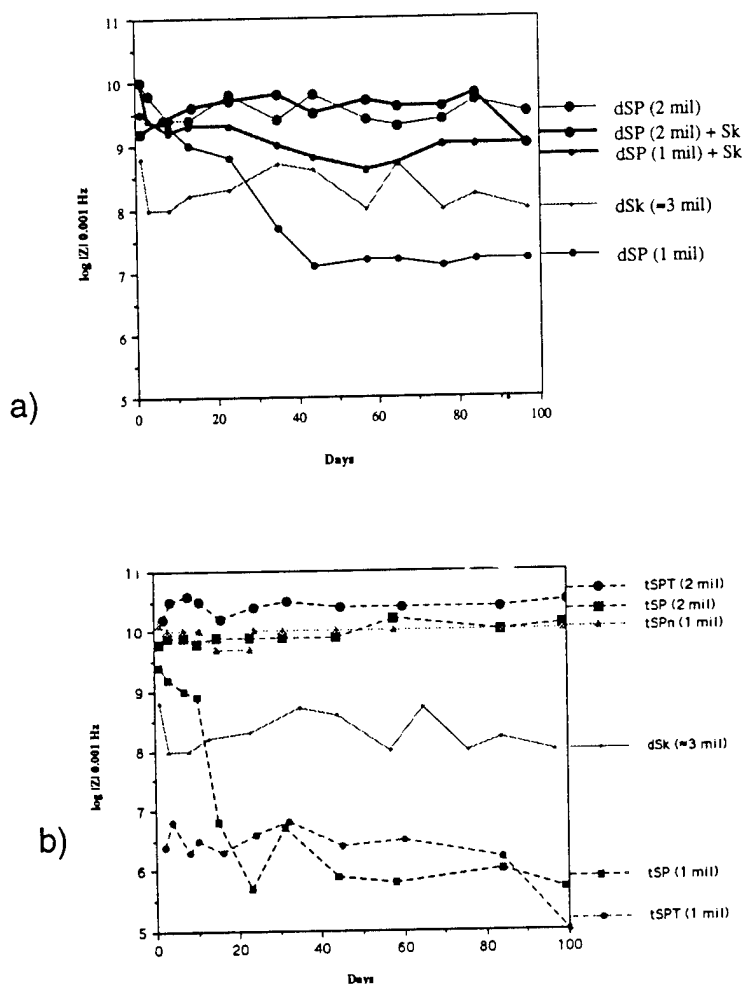


Figure 8. Efficacy of protective schemes on ZE41A with
a) Dow 17 and b) Tagnite 8200.

Figure 9 compares the impedance of the Tagnite pretreated ZE41AT5 panels with various combinations of phosphate sealant, and electrodeposited epoxy E-coat. The impedance of the E-coated samples exhibited consistently high impedance, with values above $10^{10} \Omega \cdot \text{cm}^2$, indicating excellent corrosion resistance. This Tagnite/E-coat combination clearly presents itself as an alternative scheme which may perform better than the currently used dSk system. It also shows that the impedances of the Tagnite samples, with or without a phosphate sealant, are roughly equal, indicating the apparent irrelevance of the sealant for these test conditions.

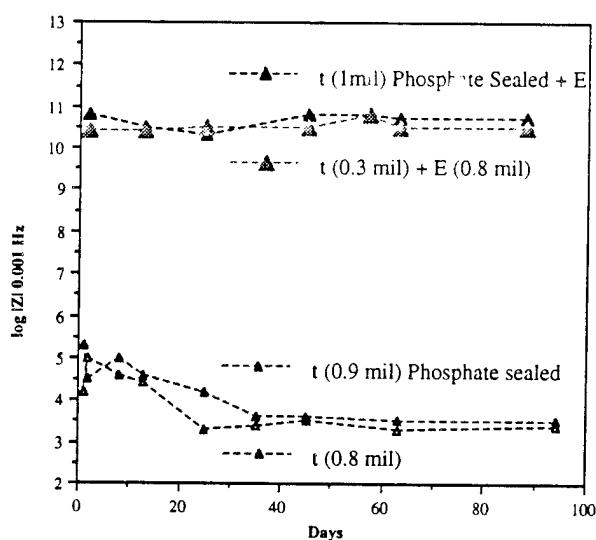


Figure 9. Effect of Tagnite 8200 on ZE41A.

It is a known fact that most of the conversion coatings such as Dow 17 which are produced by chemical immersion or anodizing are porous in nature. However, the Tagnite conversion coating layer is produced by a dual immersion and electrodeposition processes. It was reported that the pores are not continuous through the thickness of the coating layer, thus a sealant may not be required. Figure 9 shows the low impedance of Tagnite pretreated samples, $\approx 10^4 \Omega \cdot \text{cm}^2$ with or without a phosphate sealing. This fact along with the observation of gas bubbles (H_2) generated in the initial stages of the immersion test, suggest that the Tagnite conversion coating does have continuous defect pathways through to the substrate. In addition, since the sealed Tagnite performed similarly to the unsealed sample, the phosphate sealant seemed ineffective for this application. Thus a compatible sealant will definitely be required for application in a mild environment or else a sealant/primer/topcoat combination such as coating-E will be required for more

severe applications.

CONCLUSIONS

For coated ZE41A-T5 samples, there was good qualitative correlation between salt spray and EIS data. The protective schemes providing the best salt spray resistance exhibited the highest impedance values with slower rates of degradation. Impedance increases from 10^8 to $10^{10} \Omega \cdot \text{cm}^2$ indicate an upgrade in corrosion resistance from good to excellent. Several advanced protective schemes, based on either Dow 17 or Tagnite 8200 pretreatment, have shown higher impedance than the currently used coating, *provided the total thickness of the coatings is equal to or greater than 2 mils*. However, both salt spray and EIS results suggest that a 1 mil Tagnite/non-Cr primer (tSPn) coating may also provide an alternative scheme with good protection efficacy. Also, the impedance data obtained from the 1 mil Tagnite/E-Coat combination indicate that it may be another viable system. These advanced schemes should provide excellent corrosion resistance. Furthermore, the EIS testing method could also lead to a non-destructive approach to monitoring coating integrity and extrapolating the useful life of coatings. However, it should be noted that before any advanced protective schemes can be transitioned to components of operating Army aircraft, outdoor exposure testing in Army environments must be completed. In the next phase of the Army study, the degradation of these coatings during outdoor exposure studies will be monitored with a portable EIS system for correlation with laboratory test data. This information will be provided to Sikorsky Aircraft in a cooperative effort to complement their fatigue, oil exposure, strippability and touch up test program.

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